ISBN: 978-3-95450-208-0 doi:10.18429/JACoW-IPAC2019-WEPRB070

FACILE DEPOSITION OF SUPERCONDUCTING MgB_2 THIN FILMS ON SUBSTRATES: A COMPARATIVE INVESTIGATION OF ELECTROCHEMICAL DEPOSITION AND MAGNETRON SPUTTERING TECHNIQUES

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Abstract

In this work, we investigate the application of two approaches-electrochemical deposition and magnetron sputtering of MgB_2 powder, to fabricate MgB_2 films with potential accelerator applications. While electrochemical deposition yielded MgB_2 films showing superconducting properties, the adhesive strength was observed to be poor. Magnetron sputtering, on the other hand, provided uniform thin films with excellent adhesive properties but mismatch in stoichiometry meant that no superconductivity was observed. Further work to fine tune the deposition processes for large scale MgB_2 deposition inside actual RF cavities is in progress.

INTRODUCTION

Since the discovery of superconductivity in binary metallic MgB_2 (critical temperature T_c of 39K) [1], a significant amount of work has been carried out to fabricate high quality thin films of this new material for fundamental studies and device applications. One such application is the coating of Copper cavities with a layer of MgB_2 thin film, an attractive alternative to using expensive bulk Nb cavities. The advantages of such coatings include lower costs, improved thermal stability (due to the presence of Cu) as well as potential higher accelerating gradients [2].

Numerous methods have been developed over the years for fabricating MgB_2 coatings, including magnetron sputtering/co-sputtering, chemical vapour deposition, electrochemical deposition, HPCVD etc. [3-5] In this work we explore the practicality of using two different techniques, electrochemical deposition and magnetron sputtering of MgB_2 powder in an attempt to engineer stable, uniform MgB_2 films with potential superconducting properties.

MATERIALS AND METHODS

MgB_2 powder (≥99% purity, particle size: ~149 µm) was procured from M/s Sigma Aldrich and used as received. Copper (Cu) films were procured locally and cleaned thoroughly by ultrasonication in acetone bath for ~30 minutes prior to use. Si substrates were cut into small pieces of size approximately 2 × 2 cm^2 using a diamond scribe and cleaned thoroughly before use. For the magnetron sputtering deposition process, a pulsed power source (Advanced Energy Pinnacle Plus) was operated at a frequency of 350 kHz with variable input power supply.

RESULTS AND DISCUSSION

Electrochemical Deposition of MgB_2

Electrochemical deposition has previously been investigated as a technique for deposition of MgB_2 on 2D substrates [6]. However, no previous attempt has been made to use this technique for depositing onto three dimensional substrates.

MgB_2 powder (0.4 g) was dispersed in 50 ml acetone and allowed to form a homogeneous suspension by sonicating in a glass beaker for ~30 minutes, as shown in Fig. 1. Subsequently, a Cu foil of size 8 × 12 cm2 (0.1 mm thickness) was rolled to form a cylindrical tube and dipped in the beaker containing MgB_2/acetone electrolyte system. The Cu film was connected to the negative terminal of a DC power source and served as the cathode. A graphite electrode (5 mm thickness) served as the anode and was dipped in the electrolyte concentric to the Cu film (Fig. 1). The distance between the two electrodes was variable between 2 cm to 5 cm.

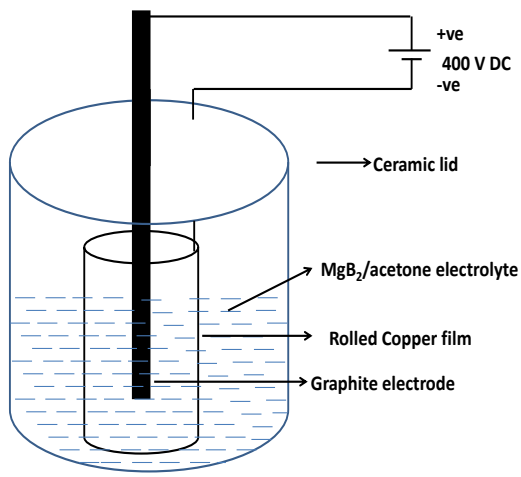

Figure 1: Set up for room temperature electrochemical deposition of MgB_2.

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Subsequently, a DC voltage of 400 V was applied between the two electrodes for different time durations ranging from two to six minutes. A thick black layer of MgB_2 was observed to form rapidly on the surface of the Cu film. The film was then subjected to post annealing in an evacuated furnace maintained at a pressure of ~10 mbar. The temperature was initially raised to 750^{0}C at a heating rate of 20^{0}C/minute, allowed to stand for 60 minutes followed by cooling back to room temperature at the same rate. Figs. 2a and 2b illustrate the DC magnetization as a function of temperature for the annealed sample and as received MgB_2 powder respectively. It can be seen that in both cases the critical transition is at 39 K; however in case of the deposited MgB_2 film, it drops gradually as compared to the as received powder where the transition is initially sharp and then followed by an almost exponentially decay. The gradual transition can be due to the formation of defects such as oxides or deviation from stoichiometry after annealing.

The presence of Mg and B in the films was also revealed by XPS analysis. However, scratch resistance tests revealed the adhesive strength of the deposited layers to be poor even after the annealing process.

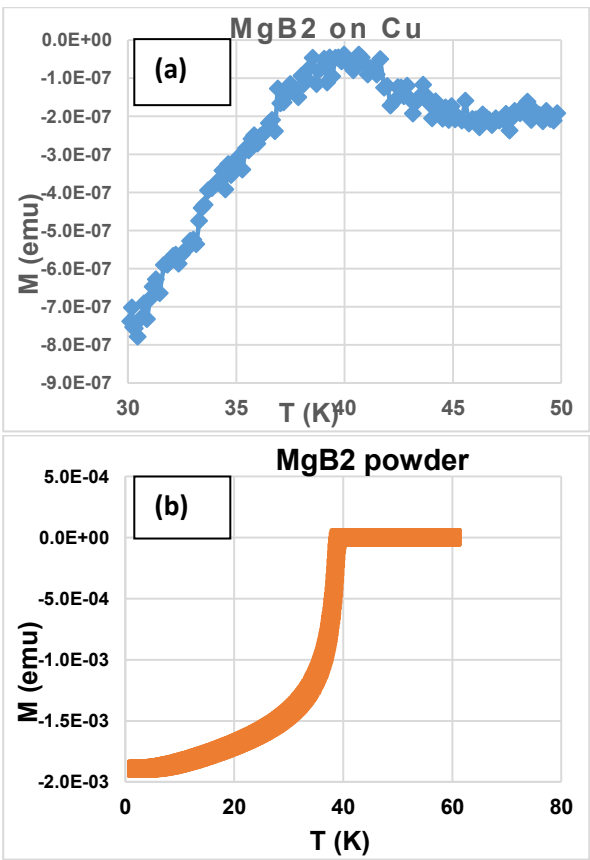

Figure 2: DC Magnetisation as a function of temperature a) MgB_2 deposited on copper and b) as received MgB_2 powder.

Magnetron Sputtering of MgB_2 Powder

Magnetron sputtering is another attractive alternative that has been explored extensively for deposition of MgB_2 films. However, few examples exist where attempt has been made to directly use MgB_2 powder as the sputtering target [7]. MgB_2 powder was spread over a shallow Cu disc. The powder was compacted to form a thin, uniform target of thickness ~3 mm. Silicon wafers of size 2 × 2 cm^2 were used as substrates for the deposition. The target to substrate distance was maintained at ~8 cm. The chamber was initially evacuated to a pressure of ~10^{-6} mbar. Operating pressure was maintained at ~4 × 10^{-3} mbar with a corresponding Argon flow rate of 5 sccm (standard cubic centimetre per minute). Pulsed DC (350 kHz, 1.1 μs) with a power of 25 W was subsequently applied for a deposition time of 4 hours. SEM-EDX analysis (Fig. 3) of the samples revealed the presence of Mg and B on the surface, with O (13.5%) and traces of Al (1.9%) as impurities. The SEM micrograph (Fig. 3a) showed a uniform, pore free and dense deposit on the substrate. However, based on the EDX analysis (Fig. 3b), the Mg/B atomic ratio calculated was lower than the desired stoichiometry of 1:2

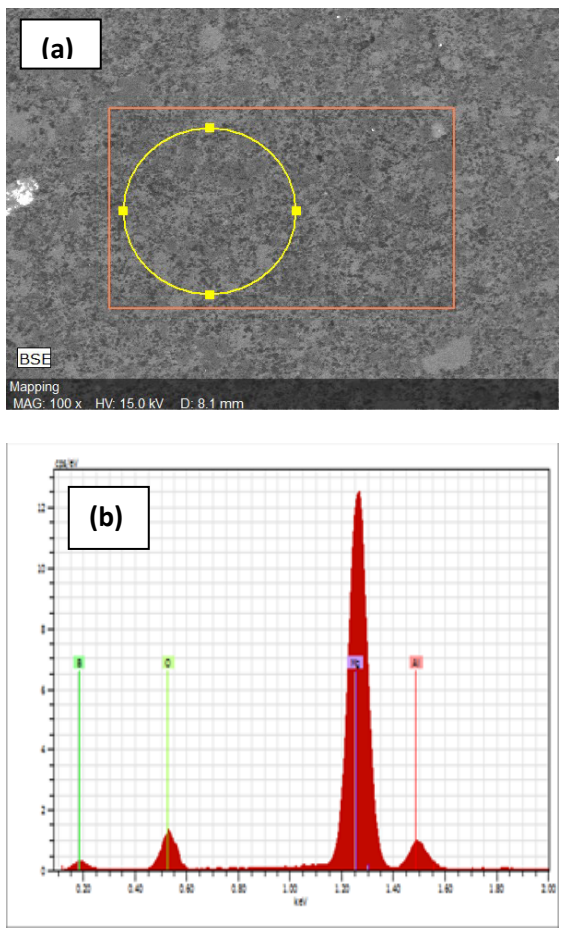

Figure 3: (a) SEM analysis of magnetron sputtered MgB_2 thin film and (b) EDX analysis MgB_2 thin film.

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This can be explained by the high volatility of Mg relative to B. In the absence of a Mg overpressure, Mg vaporization leads to a loss of Mg and mismatch in the Mg/B stoichiometry, leading to the formation of MgB_4, MgB_7 and solid B phases. Therefore, SQUID measurements of the samples did not reveal any superconducting properties in the films. However, introduction of a Mg overpressure during the deposition process and secondly, carrying out the deposition at lower powers in order to minimize the heat generated (resulting in lower rates of Mg vaporization) can address the issue of Mg loss and help maintain the desired stoichiometry.

CONCLUSION

We have explored two possible routes for deposition of MgB_2 onto substrates. Electrochemical deposition provided a rapid, facile and easily controllable route for deposition of MgB_2 on Cu films, with MgB_2 films showing a critical transition at 39 K. However, the poor adhesion of the films even after annealing process restricted large scale applicability of the process. On the other hand, magnetron sputtering of MgB_2 powder yielded uniform films with good adhesive quality. However, the poor Mg/B ratio implied that no superconducting properties were observed. Nevertheless, there is potential for further optimization and fine tuning of both processes in order to fabricate MgB_2 films with superior properties and potential superconducting applicability.

ACKNOWLEDGEMENT

This work was conducted under the aegis of the Science and Technology Facilities Council (STFC). Nilanjal Misra would also like to thank ASTEC, STFC for the support and the Rutherford International Postdoctoral Fellowship scheme for funding this work.

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